FR559A Crystal Heater

User manual 0559751

FR559 Crystal heater for use with CAD4 serial number 538-16-nn and up

For installation on KappaCCD or KappaApex systems visit http://www.bruker-axs.nl select “Support and download” click “Literature” click “Bruker AXS B.V. The Netherlands” login or register and select “KappaCCD technical user manual”.

The Crystal heater consists of:

- **Aquarium air pump**, capacity 3 litres/min. These pumps contain an electromagnet and a bellow. They operate only at a specific mains frequency, for which they have been designed. The output of a 50 Hz pump on a 60 Hz supply and vica versa is less than 50% of the nominal flow. Bruker AXS Delft supplies only pumps for 50 Hz mains supplies. Users in 60 Hz mains areas have to procure such a pump from a local aquarium (pet) shop.
- **Controller**. Check the mains voltage on the serial number label before connecting it to the local mains.
- **Eucentric goniometer head** with heater and themocouple plus a heater base for use on other goniometer heads .
- **Phi limit switch** set for use on the CAD4.

Air supply

To reduce the pulsation of the air stream from the pump, put a small glass bottle (not delivered, volume 0.3 – 0.5 litre) between the pump and the heater

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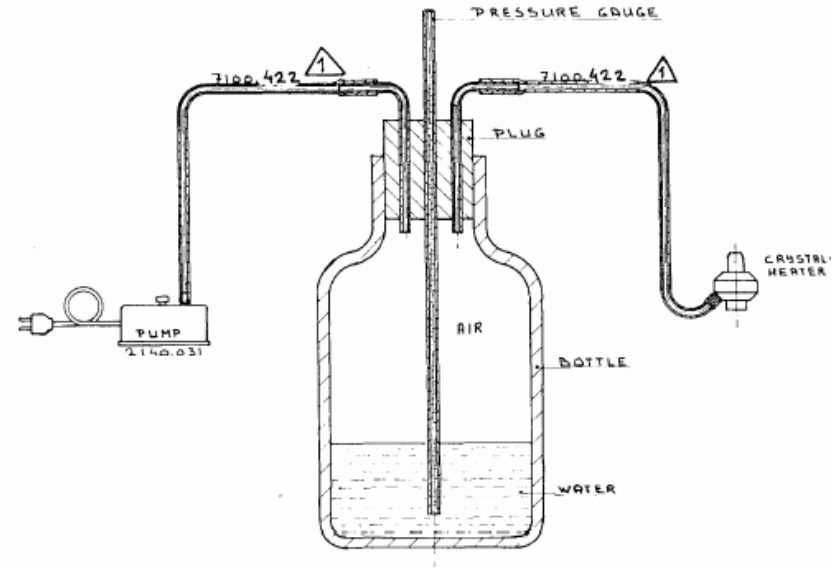


The pressure should be in the order of 50 – 100 cm water column.

Controller

All controls are located on the front of the controller. The LCD screen displays:

- The selected temperature for the experiment – switch in position “SET POINT”
- The actual temperature – switch in position “MEASURED”
- The current through the heating element – press button “HEATER CURRENT”

The green LED “SET POINT” lights up at the selected temperature.

The knob “ADJUST” sets the temperature for the experiment, on display when switch in position “SET POINT”

The red LED “ALARM” lights up when the thermocouple is disconnected or when the actual temperature and the set temperature differ too much for a too long period.

The push button “RESET” clears the electronics after the cause of the alarm has been corrected. E.g. loose electrical connections, no air, broken heater element.

The black and red connectors are the outputs to the heater element.

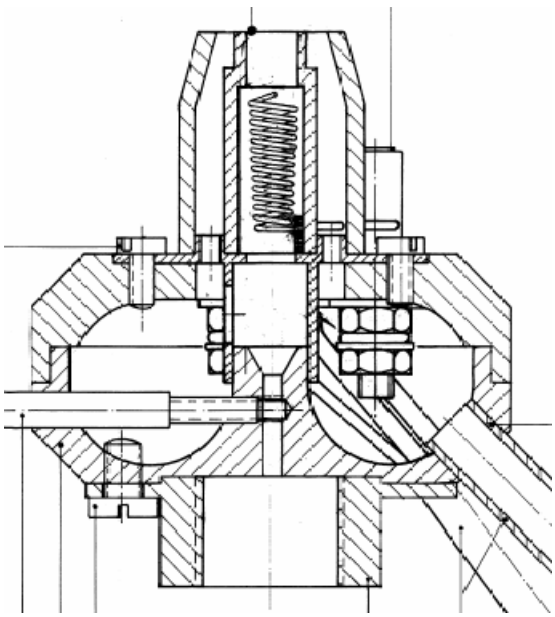
The connector “TC” is the input from the thermocouple.

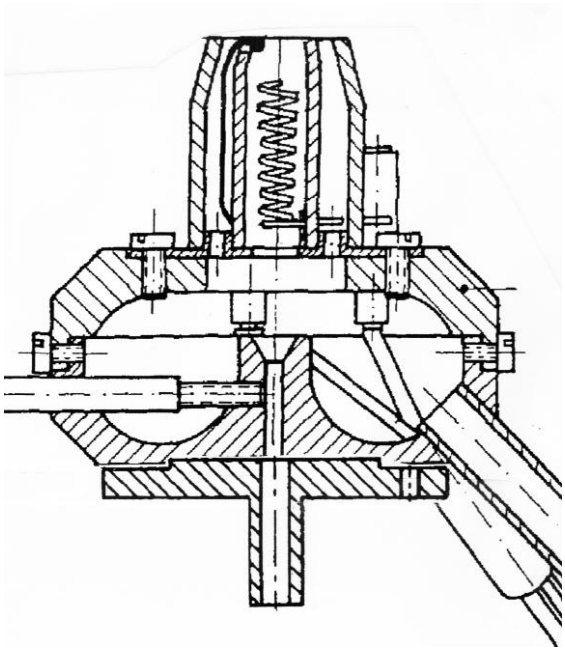
The recorder output is 1 V ≡ 100 °C.

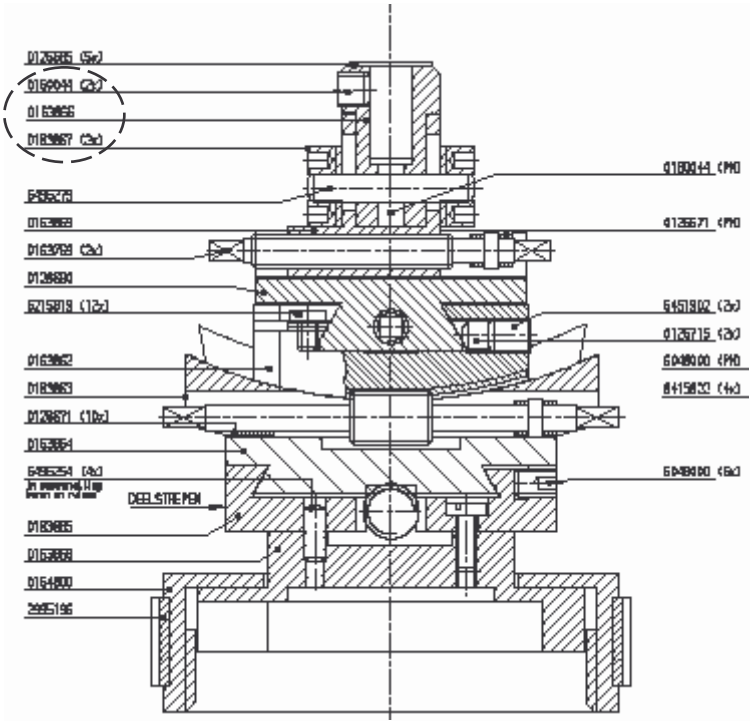
For more information about the controller, consult the service manual 0559750.

Goniometer head with heater

A special connection piece ensures a stable mounting on the eucentric goniometer head 1516918 (left). It screws on the goniometer head after removal of the upper height adjustment-locking ring and pin. The universal heater support (right) can be used with goniometer heads from other vendors.

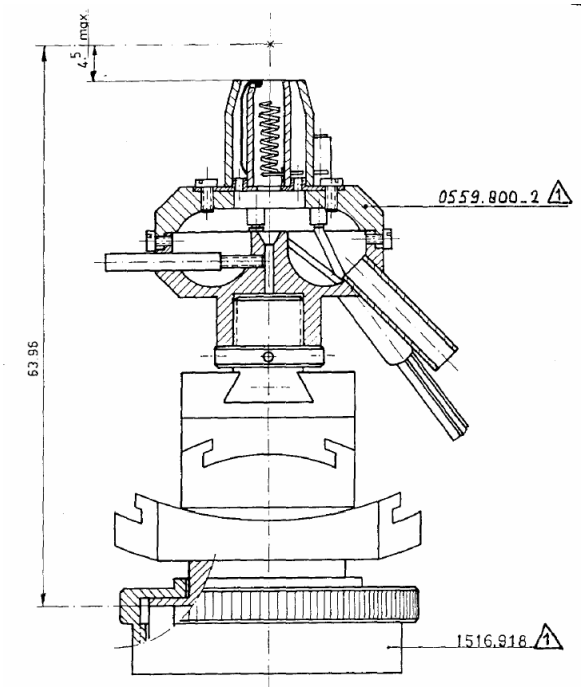






To re-build the eucentric goniometer head for regular use see the drawing on the left.

The encircled parts are packed with the spare heater elements



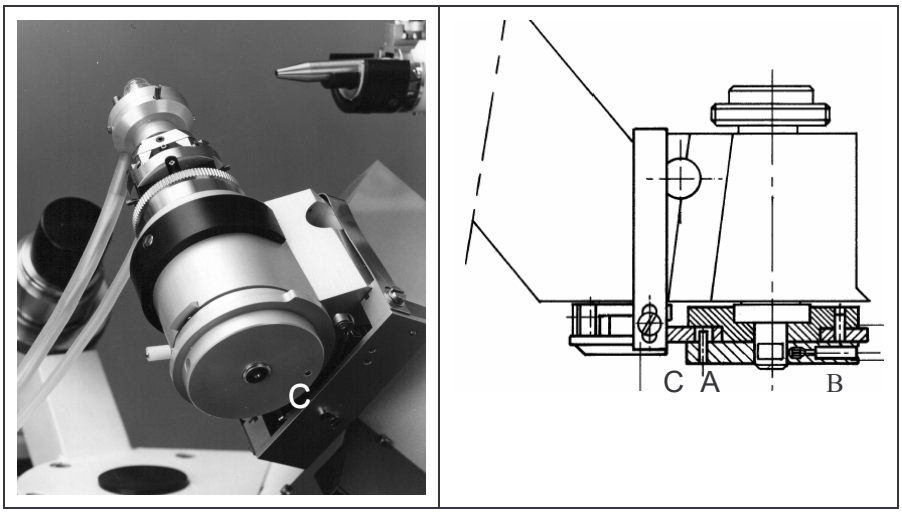
Sample position approx. 4 mm above the nozzle.

Overall height 1516.918 goniometer head with heater 63.96 mm compatible with CAD4, MACH3, KappaApex goniometers.

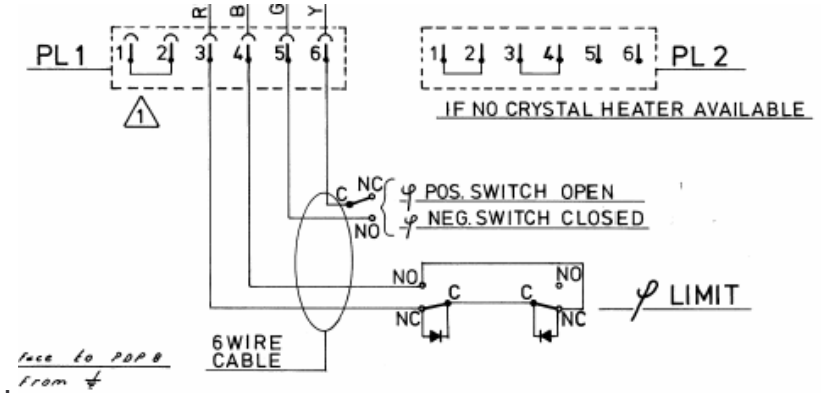
For instruments with 49 mm eucentric height use low XYZ goniometer heads.

Mounting the limit switch unit.

- Remove the Phi coarse scale disk at the back of the Kappa block. There is one screw in the centre of the block and one lateral screw.
- Remove the dripping tray at the base of the kappa support block (if present)
- Position phi to 0 degrees.
- Mount the switch unit using the support bracket and 2 screws (see picture)



- Now the disks supplied must be mounted on the phi axis; the largest one first (***phi should be at 0 degrees during this operation***).
- There is a cam in the smallest disk that fits a slit in the largest disk.(A)
- There is a Allen screw at the side of the smallest disk that can be loosened during mounting (B).
- It is important to position both disks correctly; the cam on the small disk should be close to the small vane (C) that is visible under the switch unit. The vane has 2 micro-switches that sense the left and right movement of the vane. (see scheme phi limit switch).
- Because of the cam and slit, this implies the protruding part of the biggest disk will be at the opposite side.
- Once both disks are in position and fixed to the phi axis the Allen screw can be tightened (B).
- In the CAD4 program go to CASPAR and set "phi limit switch present".
- The top angle of the goniometer head with the heating device is 90° and the maximum radius to 30 mm. Do not use larger goniometer heads to prevent crashes outside the regular collision collars.
- Fix the connector of the switch unit to the socket marked "Limit switch" at the back of the goniometer. During standard use a dummy plug is in this socket; it must be put back after removing the limit switch unit.



Phi limit switch diagram

- Enough slack should be kept for the several cables running between the limit switch unit, the heater and the outside world, also when moving omega and kappa.
- Restart the CAD4 program. Check the proper operation of the limit switch without the heater. The phi range is now limited to -240°<phi<+240°. Position phi = 180 and from that position to phi = 270. With the new set up phi should go to 270 via 0.
- When not OK restart the CAD4 program a second time before starting to correct the hardware installation.

Glue

The sample is fixed on a quartz fibre and the fibre is attached to a metal pin.

- Sample to fibre bond:
  Cerastil C-3 (1100 ºC) or C-10 (1650 ºC) (www.panacol.de) or a 50 / 50 % mix of waterglass and manganese oxide (800 ºC) can be used to fix the sample to the quartz fibre.
- Fibre to metal pin bond:
  Torr Seal Resin Sealant, Varian part number 9530001 can be used for the quartz-metal bond. (www.varianinc.com temperature range –45 ºC to 120 ºC)

Temperature calibration

See the original publication by F. Tuinstra and G.M. Fraase Storm J. Appl. Cryst. (1978), **11**, 257-259
Some older literature about HT experiments mentions mounting crystals of known melting range and observing the start of the melting with the aid of a microscope.
Crystals used were:

Diphenylacetylene	m.p. 61-62 ºC
Urea	132.5-134.5 ºC
Thiourea	171-171.8 ºC
$AgNO_3$	212 ºC
$NaNO_2$	271 ºC
$NaNO_3$	307 ºC
KNO_2	440 ºC
NaCN	562 ºC
NaI	650 ºC